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NASA TECHNICAL MEMORANDUM

A COMPOSITE SYSTEM APPROACH TO AIRCRAFT CABIN FIRE SAFETY

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16. Abstract

The thermochemical and flammability characteristics of two polymeric composites currently in use and seven others being considered for use as aircraft interior panels are described. The properties studied included: (1) limiting oxygen index of the composite constituents; (2) fire containment capability of the composite; (3) smoke evolution from the composite; (4) thermogravimetric analysis; (5) composition of the volatile products of thermal degradation; and (6) relative toxicity of the volatile products of pyrolysis. The performance of high-temperature laminating resins such as bismaleimides is compared with the performance of phenolics and epoxies. The relationship of increased fire safety with the use of polymers with high anaerobic char yield is shown. Processing parameters of one of the bismaleimide composites is detailed.

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detailed.

INTRODUCTION

The purpose of this program was to assess the relative flammability and
thermochemical properties of some typical state-of-the-art and candidate
experimental aircraft interior composite panels, and to develop an understand-
ing of the relationship of flammability and thermochemical properties of these
systems.

Composite sandwich panels constitute most of the surface of aircraft
interiors as sidewalls, partitions, ceiling panels, and overhead stowage bins.
Currently used composites meet or exceed regulatory requirements [1] and offer
excellent aesthetic, serviceability, maintenance, and other properties. How-
ever, additional improvements are being sought to reduce ignition susceptibil-
ity, fuel contribution, smoke and toxic fume emission, and to increase fire
containment capability [2-8]. Experimental composite panels that could offer
improved fire resistance and smoke reduction in aircraft fires are now being

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developed and tested. In this program, nine different types of experimental composite panels were evaluated in terms of their flammability properties. Two of these were typical state-of-the-art interior panels and seven were experimental. The composite panels used by most airframe manufacturers as interior paneling are sandwich panels that vary slightly in configuration, component composition, thickness, and density depending on the type of aircraft in which they are used and the specific application. In general, the panel consists of polyvinyl fluoride decorative finish bonded to a fiberglass-resin laminate which is bonded to an aromatic polyamide honeycomb core as shown in Figure 1.

DISCUSSION

Description of Composites

Nine types of composite panels were evaluated. Three types of resin systems were used for the fabrication of the laminates used in these composites: epoxy, bismaleimide, and various modifications of phenolic resins. The general chemical structure of these resins is shown in Figure 2; the composition of the composites is shown in Tables 1-3. All composites fabricated were 2.54 cm thick. Composite No. 1-6 had a decorative surface of a polyvinyl fluoride film printed with an acrylic ink and bonded to a laminate. The laminates consisted of various types of fiberglass preimpregnated with various types of phenolic resins. The laminates were adhered to the hexagonal-cell aromatic polyamide honeycomb structure using various types of phenolic resin-fiberglass adhesive ply. Composite No. 1 was considered a typical state-of-the-art phenolic resin panel. Resins used in the preparation of the laminates for composites No. 1-6 were obtained commercially and are designated as phenolic types A through G. The exact formulation for these resins was not available

from the suppliers. The decorative laminates of composites No. 1-6 were press-bonded to the honeycomb using an adhesive bond ply at 160° C for 12 min at 689.6 kN/m² pressure. The sandwich panel was then cured at 123° C for 1 h with 50 mm Hg minimum vacuum bag pressure. Composite No. 7 was composed of a laminate of bismaleimide-fiberglass adhered to the aromatic polyamide honeycomb. The processing and composition of this type of composite was similar to those of the advanced composite panel described previously [9] except that a foam produced by heating quinone dioxime was used as the core filler material [10]. Composite No. 8 is a typical state-of-the-art composite panel consisting of polyvinyl fluoride film bonded to a laminate of epoxy-fiberglass. The laminate is adhered to the aromatic polyamide honeycomb using an epoxy-fiberglass ply. Process description and composition of this composite has been described in detail previously [9,11,12]. Composite No. 9 was similar to composite No. 7 except that a bismaleimide-fiberglass honeycomb is used that is partially filled with a mixture of carbon microballoon and bismaleimide resin.

Processing and Fabrication, Composite No. 9

The fabrication process for composite No. 9 is shown schematically in Figure 3. Processing a typical 30 cm × 30 cm × 2.5 cm composite panel of this type is done as follows.

FACE SHEET FABRICATION PROCESS.— The face sheet fabrication essentially consists of resin preimpregnation of the fiberglass (MIL-C-9084C Type IIX) cloth, and the forming and cure of the prepreg into the required face sheets.

A 50% solution is prepared by sifting bismaleimide resin powder into N-methyl-2-pyrolidone solvent under vigorous agitation, avoiding resin caking on the container walls or lump formation; a high-speed agitator is used. The initial viscosity of a 50% by weight solution is less than 10 P at room

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temperature, but the viscosity rapidly increases with aging. Solutions are prepared immediately prior to use. Under controlled storage conditions, a maximum use-life of 20 days is projected for the prepared resin solution.

The process used to impregnate the resin into the fiberglass consists of passing the dried cloth through the 50% solution of bismaleimide resin. Further resin impregnation and removal of excess resin is accomplished by passing the resin wet cloth between a steel roller and a wiper blade.

To achieve uniform impregnation, the glass fabric is pulled through the impregnator at a constant speed of 0.6 m/min with a constant wiper blade pressure exerted on the impregnated cloth. The prepregged cloth is then B-staged in an air-circulation oven for 15 min at 82° C and for 30 min at 93° C. The prepreg thus processed has average resin content and volatile content of 41.3% and 5.2%, respectively.

The prepreg face sheets are cured using either the autoclave vacuum bag technique or platen pressure method. In the autoclave vacuum bag technique, a 33 cm × 33 cm prepreg single layer cloth sandwiched between porous teflon-coated glass fabric is placed on a 0.6-cm-thick aluminum plate. On top of this, a glass bleeder cloth is placed against the sandwiched prepreg. This assembly is vacuum bagged and cured at an external pressure of 345 kN/m^2 and a temperature of 177° C for 1 h. In the press cure method, the 35 cm × 35 cm prepreg material is sandwiched between porous teflon-coated fabric and cured between 0.6-cm-thick aluminum plates treated with a mold release. The prepreg is cured at 345 kN/m^2 for 1 h at 177° C. After cure, the face sheets are visually checked for flaws, voids, thickness uniformity, and resin content. Thickness of the face sheets fabricated averaged 0.025 cm and the sheets contained 30% to 34% resin.

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CORE FABRICATION PROCESS.— The core consists of a bismaleimide-fiberglass honeycomb filled with carbon microballoons bound with bismaleimide resin. The carbon microballoons are prepared by pyrolyzing phenolic microballoons in a nitrogen atmosphere. A stainless steel container is filled with phenolic microballoons and enclosed in a larger stainless steel container with a nitrogen inlet to provide an oxygen-free atmosphere. The assembly is placed in a larger furnace. The pyrolysis cycle is as follows: room temperature to 816° C in 4 h; hold at 816° C for 4 h; and cool to room temperature in 2 days. Pyrolyzed carbon microballoons must be cooled to 38° C, before removal of the nitrogen blanket, to prevent spontaneous ignition of the carbon microballoons. After pyrolysis the carbon microballoons are no longer free-flowing and are agglomerated as large cakes. To break them into smaller agglomerates, the caked microballoons are placed in a container with isopropanol (ratio of 1 kg balloons/7 liters solvent) and mixed in a paint shaker for 15 min. The slurry is then screened through a 20-mesh screen to remove the larger non-separated agglomerates. The screened isopropanol/carbon microballoon slurry is now ready for core impregnation.

The equipment illustrated in Figure 4 is used to fill the cores of the fiberglass-bismaleimide honeycomb with the prepared carbon microballoons. A high density 0.3-cm cell aluminum honeycomb is fitted and restrained on the bottom inside of the vacuum filling box. A nylon screen (120 mesh) is placed between the aluminum support honeycomb and the fiberglass reinforced polyimide honeycomb to retain the microballoons. High vacuum is not required to effectively impregnate the honeycomb, but a high volume of air displacement is required. A vacuum reservoir chamber is pumped to a vacuum of approximately 10 mm Hg.

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To make honeycomb that is completely filled with the microballoons, the carbon microballoon/isopropanol slurry is poured over the honeycomb. To control the fill rate, the vacuum applied is regulated by the valve between the box and solvent recovery trap. The negative pressure causes the microballoons to be pulled into the cells. This process is repeated several times to fill all cells. Excess slurry is then removed with a teflon blade.

To make honeycomb that is partially filled with a particular quantity of microballoons, the following method is used: Prior to placing the fine mesh screen and the fiberglass-bismaleimide honeycomb into the vacuum filling box, the vacuum control valve to the box is closed. The vacuum filling box is filled with isopropanol to the top edge of the aluminum support honeycomb. After placement of the fine mesh screen and the fiberglass bismaleimide honeycomb, the isopropanol-microballoon slurry with the desired microballoon weight content is poured over the honeycomb. Sufficient isopropanol is used to achieve good flow and complete coverage of the honeycomb. The vacuum control valve is then opened to draw the microballoons into the cells, and the s 'vent is removed and contained in the solvent recovery trap. The filled honeycomb cores, sandwiched between two nylon, fine-mesh screens and between two aluminum support honeycombs, are dried for 16 h in an air-circulating oven at 93° C. After drying, the microballoon fill is saturated with a 2% solution of bismaleimide resin in N-methyl-2-pyrolidone solvent. On cores that are partially filled with the microballoons, the 2% solution is sprayed into the cores at low air and material pressure. On cores that are completely filled with microballoons, the spray process will tend to blow out the microballoons; therefore, the 2% solution is applied by brush. Care must be exercised to uniformly maintain the filled microballoons within the cells. After saturation

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of the microballoons with the 2% bismaleimide resin solution, the honeycomb is heated for 2 h at 93° C and for 1 h at 204° C to completely cure the bismaleimide binder. Prior to final assembly, the combined weight of the microballoons, bismaleimide resin binder is checked. For the microballoon resin combination, the resin by weight is approximately 4-10%. For a half-filled 2.4-cm-thick core, the fill weight should be approximately 145 g/1000 cm^2.

SANDWICH STRUCTURE PANEL ASSEMBLY.— The assembly process consists of forming the aircraft interior panel by bonding the face sheets to the microballoon filled fiberglass bismaleimide honeycomb panel with a polyimide film adhesive.

Prior to the bonding operation, the face sheets and the microballoon-filled honeycomb are cleaned. To achieve the required fileting of the film adhesive to the core, loose microspheres must be removed from the bonding faces and the faces cleaned with a methyl-ethyl-ketone-soaked cleaning cloth. The assembly is then placed in a platen press at 204° C and cured for 2 h at 700 kN/m^2. Afterward, the panel is cured for 24 h at 254° C to remove volatile materials and to achieve reduced smoke characteristics.

On completion of the assembly, the panel is weighed and examined for uniformity of bonding.

TEST RESULTS AND ANALYSIS

Thermochemical Characterization of Composites

Samples of the nine types of composites were cut to a size of 2.5 cm × 2.5 cm × 2.5 cm and were ground uniform to approximately 250 mesh. The samples were subjected to the following thermochemical studies in order to (1) determine the relative thermal stability of the samples under anaerobic and oxidative conditions, (2) determine the major volatile products produced

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from the pyrolysis of the samples in vacuum, and (3) determine the relative
toxicity of the pyrolysis effluents by exposing animals to them.

Thermogravimetric Studies

Thermal analyses of the composites were conducted on a DuPont 950 Thermo-
gravimetric Analyzer (TGA) using both nitrogen and air atmospheres with a
sample size of 10 mg. The thermogravimetric analyses data of 10° C/min heating
rate in nitrogen and in air are shown in Figures 5 and 6, respectively.

The pyrolysis of the samples in air and nitrogen atmospheres was conducted
to obtain a relative understanding of the pyrolysis of the samples in the fur-
nace used to pyrolyze samples for assessing their relative toxicity as
described later in the text. Pyrolysis in an air atmosphere is intende. to
approximate the environment in the pyrolysis tube at the start of the toxicity
test, and pyrolysis in a nitrogen atmosphere is intended to approximate the
environment in the pyrolysis tube during the test after the original air has
been essentially displaced by pyrolysis effluent. The degradation products are
continuously removed from the sample during thermogravimetric analysis, and in
the relative toxicity test apparatus described later they are conveyed only by
normal thermal flow. The TGA data in the nitrogen atmosphere are considered
more relevant, because in the toxicity apparatus the pyrolysis effluents that
evolved at lower temperature have essentially displaced the original air by the
time the temperature has reached 700° C.

Composite No. 9 is the most stable composite and gives the highest char
yield in nitrogen. All the composites except No. 7 were oxidized completely
in air above 600° C and gave constant weight residues.

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Analysis of Volatile Products

Samples of the composites were pyrolyzed using the apparatus shown in Figure 7. The samples were placed in quartz tubes that were 2.5 mm in diameter. Each sample tube was attached to a manifold and evacuated to 10^{-4} torr. There was a stopcock between the manifold and the sample tube so that the sample tube could be isolated while gas samples were being collected. An infrared cell, attached to the manifold via a stopcock, a mercury manometer, and a trap were also attached to the manifold. At the beginning of a pyrolysis run the stopcock to the vacuum pump was closed and a furnace at 700° C was placed around the sample tube. At this point a timer was started. The pressure of the gases evolved during the pyrolysis was monitored with the pressure gauge. After 5 min the furnace was removed, the stopcock to the sample tube was closed, and the stopcock leading to the infrared cell was opened allowing the pyrolysis gases to enter the infrared cell. After a pressure reading was taken, the stopcock leading from the infrared cell to the gas manifold was closed. Dry air was admitted to the infrared cell so that the total pressure was equal to atmospheric pressure. This was done so that the pyrolysis gases were always measured at the same total pressure, the main portion of which was dry air, thus eliminating the effects of pressure broadening. Infrared spectra were taken using a Perkin Elmer Model 180 infrared spectrometer. Finally, the sample tube was removed from the manifold, broken open, and the residual char was weighed.

Part of the material that was volatile at 700° C condensed on the sample tube as it was removed from the furnace; salt plates were made from methanol solutions of this material. Infrared spectra, obtained from the salt plates, appeared to be those of a mixture. All infrared spectra displayed a sharp

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intense line at 2240 cm^{-1}. This type of an absorption is characteristic of organic compounds containing a -CN group.

The results of the analysis of the volatile species are presented in Tables 4 and 5. These results were obtained from samples that were pyrolyzed in vacuum. A considerably different distribution of products might have been obtained had the samples been pyrolyzed in air, in which case the products would be a function of the partial pressure of oxygen at the sample, the temperature of pyrolysis, and the time that it took the sample to reach the pyrolysis temperature.

Flammability Properties

THERMAL EFFICIENCY.— The NASA Ames T-3 thermal test (Fig. 6) [13] was used to determine the fire endurance or fire containment capability of the composite panels. In this test, specimens measuring 25 cm × 25 cm × 2.54 cm thick are mounted in the chamber and thermocoupled on the backface of the specimen. The flames from an oil burner supplied with approximately 5 lit/h of JP-4 jet aviation fuel provide heat flux to the front face of the sample in the range of 10.4-11.9 W/cm^2.

The fire endurance capability of the nine composite panels is compared in Figures 7-9. In these figures, the backface temperature rise of the panel is plotted as a function of the time in minutes when the sample is subjected to this type of fire. It can be seen (Fig. 8) that the backface temperature of the conventional composite, No. 8, reached 200° C in 2.5 min whereas it took as long as 8 min for the bismaleimide composites, No. 7 and No. 9, to reach a comparable backface temperature.

LIMITING OXYGEN INDEX.— The limiting oxygen index (LOI) of the components comprising the composites was determined per ASTM D-2863 and are indicated in

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Table 6. The values indicated are for the laminated or composite components as they are used in the sandwich composite and not for the individual polymers. The average LOI value shown for each composite structure is based on the values obtained from the components.

SMOKE EVOLUTION.— Smoke evolution from the composites was determined using NBS-Aminco Smoke density chambers at two laboratories: laboratory A and laboratory B. The procedure and test method used were essentially those described by NFPA 258-T [14]. A detailed description of the NBS smoke chamber can be found in Reference 15.

The test results obtained with the NBS smoke chamber, modified by the incorporation of an animal module accessory [16], are presented in Table 7. Values of specific optical density (Ds) at 1.5 min, 4.0 min, and maximum (Dm) are presented; standard deviations are also given.

Composites No. 1 and 8 represented the state-of-the-art baseline materials. All the other composites exhibited significantly lower smoke density values, indicating that the phenolic and bismaleimide offer the advantage of smoke reduction.

A comparison of the Ds values obtained by the two laboratories is presented in Table 8. In addition to possible differences in apparatuses at the two laboratories, the calculation procedures were slightly different. In laboratory A, the Ds values are obtained from individual test data and then averaged. In laboratory B, an average curve is generated by computer from the data of the individual tests, and the Ds values are obtained from the computer-averaged curve. The smoke density of composite panels similar to composite No. 8 has also been evaluated by other laboratories [17]. Composition of the

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panel was essentially the same as composite No. 8 except the panel was 0.70 cm thick. The maximum smoke level, Dm(corr) was 54 whereas the average in the present studies was Dm 58.7.

RELATIVE TOXICITY.— Efforts to obtain relative toxicity information by using the NBS smoke chamber with the animal module accessory were unsuccessful. The mice and rats exposed during the standard smoke tests showed no evidence of death or even incapacitation [15]. The heat flux of 2.5 W/cm^2 used in the standard test procedure appears to be incapable of producing sufficient effluents from these high-performance materials.

To provide an indication of relative toxicity, 1.0 g of each of the powdered specimens of the composites was pyrolyzed as a heating rate of 40° C/min in a quartz tube to an upper temperature limit of 700° C, and the effluents conveyed by natural thermal flow into a 4.2 liter hemispherical chamber containing four Swiss albino male mice. The apparatus (Fig. 12) and procedure have been described previously in detail [18,19]. The test was continued for 30 min, unless terminated earlier upon the death of all four animals. The highest chamber temperature recorded was 29.5° C, indicating that the pyrolysis gases were adequately cooled before entering the animal exposure chamber. Some condensation of higher-boiling vapors in the connecting tube was observed, and some of the effluent gases entered the animal exposure chamber as visible heavy vapors, indicating that some higher-boiling compounds did reach the animals and were not lost entirely by cooling. The lowest oxygen concentration recorded was 12%, indicating that hypoxia was not a significant factor in animal response. Table 9 shows the relative toxicity to mice of the degradation products from the powdered composites when heated in this manner.

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During the 30-min exposure period, composite No. 9 caused no deaths in the test animals. The other composites, that is, No. 1-8 caused death to the animals at times ranging from 19.65 min to 28.31 min.

The test time-to-death was judged as the time elapsed at cessation of movement and respiration, as judged by the observer. Time to incapacitation was judged as the time to the first observation of loss of equilibrium, collapse, or convulsions, whichever came first. As a comparison, 1.0 g of wool fabric causes death to four mice in approximately 9.5 min when tested in a similar manner.

EFFECT OF CHAR YIELD ON SMOKE EVOLUTION AND OXYGEN INDEX.— Previous studies [20] have shown a correlation between the flammability properties of polymers and their char yield. A decrease in ease of ignition and smoke evolution was observed with high char yield polymers. The same relationship seems to exist with composites consisting of polymers and inorganic reinforcements.

Figure 13 compares the smoke density and relative anaerobic char yield of those composites when they are tested in the NBS smoke chamber. It can be seen that in general composites with high char yield have fairly low smoke evolution.

Figure 14 compares the limiting oxygen index of these composites with their relative anaerobic char yield. It can be seen that in general composites with very high char yield exhibited a high limiting oxygen index.

Thermophysical Characterization of Composites

The thermal conductivity of panels No. 8 and No. 9 were determined in accordance with ASTM C-177-45. The thermal conductivity of composite No. 8 was significantly higher than that of composite No. 9, probably due to the absence of any insulative material in the honeycomb; the data are presented in Table 12. The flatwise tensile strength of the laminates to the honeycomb was

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determined per ASTM C-307; as shown in Table 10, composite No. 9 has a slightly lower tensile strength than the state-of-the-art composite No. 8. In addition to these properties, the flammability properties of these composites are compared in Table 10.

CONCLUSIONS

Composite No. 9, consisting of bismaleimide-fiberglass/bismaleimide honeycomb with carbon microballoons, exhibited the highest fire containment capability. Similarly, composite No. 7 exhibited high fire containment capability.

Advanced composite panels consisting of PVF/phenolic-fiberglass/aromatic polyamide honeycomb/phenolic-fiberglass (composites No. 2-6) and composites No. 7 and 9 exhibited lower smoke evolution than the state-of-the-art composite No. 8.

The relative toxicity of the pyrolysis products of composite No. 9 was the lowest, as measured using the methodology indicated, of all the composites tested. It had also the highest anaerobic char yield of all the composites tested.

A correlation was established between the anaerobic char yield of the composites and their relative limiting oxygen index and smoke evolution. Generally, composites consisting of polymers with high anaerobic char yield, had a high limiting oxygen index and low smoke evolution.

No definite correlation was found between the concentration of the toxic pyrolysis products of the composites and their relative toxicity to animals indicating possibly that additional toxic species may be present both in the volatile gases, which accounted for only 18% of the degradation products, and in the solid particulates. Additional studies should be conducted using gas

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chromatography-mass spectometry to identify these compounds and their relative concentrations.

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for TRW, Cleveland, Ohio and General Electric Company, Coshocton, Ohio. Since 1966 Mr. Gilwee has been working on the development and processing of high temperature polymers and fire retardant materials. He is a member of the American Chemical Society and the Society of Plastics Engineers.

N. R. Lerner

N. R. Lerner is a research scientist in the Chemical Research Projects Office of NASA at the Ames Research Center at Moffett Field, California. She received her B.A. degree in chemistry, cum laude, from Hofstra University in 1956; her M.S. degree (1959) and Ph.D. degree (1962) were received from the University of Chicago. Since joining NASA in 1970 she has worked on flame inhibition and on mechanisms of thermal degradation of polymers.

Carlos J. Hilado

Carlos J. Hilado is a research professor in the Institute of Chemical Biology of the University of San Francisco, and conducts research programs at San Francisco and at the NASA Ames Research Center at Moffett Field, California. He received his B.S. degree in chemical engineering from De La Salle College in Manila, Philippines in 1954; his M.S. degree in chemical engineering practice from Massachusetts Institute of Technology in 1956; and his Sc.D. degree in applied science, honoris causa, from De La Salle University in 1974. His teaching experience includes De La Salle College and the West Virginia College of Graduate Studies. From 1956 to 1975 he was a member of the Chemicals and Plastics Research and Development Department of Union Carbide Corporation at South Charleston, West Virginia. He has had experience in sugar milling and refining, food technology and microbiology, pulp and paper,

vinyl and urethane foams, vinyl polymerization, materials evaluation, industrial insulation, and fire safety research.

Lisa A. LaBossiere

Lisa A. LaBossiere is a technical assistant in the Institute of Chemical Biology of the University of San Francisco. She received her B.A. degree in psychology from Boston College in 1976. Before starting work on toxic materials characterization, she worked in the Department of Pathology at Harvard Medical School, Department of Neuroscience at Children's Hospital in Boston, and Department of Psychology at Boston College. She has had experience in animal care and breeding, tissue culture, animal immunization, and activity studies in stressful environments.

M.t. Hsu

Ming-ta S. Hsu is a lecturer in the Department of Chemistry, San Jose State University at San Jose, California. She received her B.S. degree in chemistry from National Taiwan University in 1960, her M.S. degree in chemistry from New Mexico Highlands University in 1963, and her Ph.D. degree in chemistry from Iowa State University in 1967. Before joining the San Jose State University in 1972, she was a NRC postdoctoral associate with NASA-Ames Research Center. She has worked on characterization and thermal degradation of polymers.

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TABLE 1.- COMPOSITION OF COMPOSITES: NO. 1-3.

	COMPOSITE NO.	1	2	3
A	DECORATIVE SURFACE, cm THICK, % WT	0.002 PVF CLEAR ACRYLIC INK, 0.005 PVF	SAME AS 1-A	SAME AS 1-A
B	FACE SHEET, RESIN/FABRIC: % WT	PHENOLIC TYPE A/7581 GLASS	PHENOLIC TYPE C/7581 GLASS	SAME AS 2-B
C	BOND SHEET, RESIN/FABRIC: % WT	PHENOLIC TYPE B/120 GLASS	PHENOLIC TYPE D/120 GLASS	PHENOLIC TYPE C/120 GLASS
D	CORE TYPE; THICKNESS, cm; CELL SIZE, cm; DENSITY, kg/m^3	AROMATIC POLYAMIDE-PAPER HONEYCOMB; 2.413; 0.31; 48.06	SAME AS 1-D	SAME AS 1-D
E	CORE FILLER; DENSITY, kg/m^3	NONE	NONE	NONE
F	SAME AS C	SAME AS 1-C	SAME AS 2-C	SAME AS 3-C
G	SAME AS B	SAME AS 1-C	SAME AS 2-C	SAME AS 3-C
H	SAME AS A	NONE	NONE	NONE
	COMPOSITE DENSITY, kg/m^3	72.410	79.138	70.488

TABLE 2.- COMPOSITION OF COMPOSITES: NO. 4-6.

	COMPOSITE NO.	4	5	6
A	DECORATIVE SURFACE, cm THICK; % WT	0.002 PVF CLEAR ACRYLIC INK 0.005 PVF	SAME AS 4-A	SAME AS 4-A
B	FACE SHEET, RESIN/ FABRIC; % WT	PHENOLIC TYPE E/ 7581 GLASS	PHENOLIC TYPE F/7581 GLASS	PHENOLIC TYPE G/7581 GLASS
C	BOND SHEET, RESIN/ FABRIC; % WT	SAME AS 4-B	PHENOLIC TYPE F/120 GLASS	PHENOLIC TYPE G/120 GLASS
D	CORE TYPE; THICKNESS, cm; CELL SIZE, cm; DENSITY, kg/m³	AROMATIC POLYAMIDE-PAPER HONEYCOMB; 2.413; 0.31; 48.06	SAME AS 4-D	SAME AS 4-D
E	CORE FILLER; DENSITY, kg/m³	NONE	NONE	NONE
F	SAME AS C	PHENOLIC TYPE E	SAME AS 5-C	SAME AS 6-C
G	SAME AS B	SAME A: 4-F	SAME AS 5-C	SAME AS 6-C
H	SAME AS A	NONE	NONE	NONE
	COMPOSITE DENSITY kg/m³	76.575	76.095	70.968

TABLE 3.- COMPOSITION OF COMPOSITES: NO. 7-9.

	COMPOSITE NO.	7	8	9
A	DECORATIVE SURFACE cm THICK; % WT	NONE	0.002 PVF ACRYLIC INK 0.005 PVF	NONE
B	FACE SHEET, RESIN/FABRIC; % WT	BISMALEIMIDE/120 GLASS	EPOXY TYPE H/181 E GLASS B + C = 35.9%	BISMALEIMIDE/181 E GLASS B + G = 14.1%
C	BOND SHEET, RESIN/FABRIC; % WT	POLYIMIDE ADHESIVE	EPOXY TYPE H/120 GLASS	SAME AS 7-C C + F = 5.1%
D	CORE TYPE: THICKNESS, cm; CELL SIZE, cm; DENSITY, kg/m³, % WT	AROMATIC POLYAMIDE-PAPER HONEYCOMB: 2.413; 0.31; 48.06	SAME AS 7-D 2.413; 0.31; 48.06; 20.5%	BISMALEIMIDE-GLASS HONEYCOMB: 2.413; 0.47; 80.1; 30.3%
E	CORE FILLER: DENSITY, kg/m³, % WT	QUINONE DIOXIME FOAM	NONE	CARBON MICRO-BALLOONS WITH 5% BISMALEIMIDE; 112; 50.5%
F	SAME AS C	SAME AS 7-C	SAME AS 7-C F + G = 35.9%	SAME AS 7-C
G	SAME AS B	SAME AS 7-B	SAME AS 7-B	SAME AS 9-B
H	SAME AS A	NONE	SAME AS 7-A A + H = 7.7%	NONE
	COMPOSITE DENSITY, kg/m³	110	95	130

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TABLE 4.- MAJOR VOLATILE PRODUCTS AT 23° C FROM THE PYROLYSIS OF COMPOSITES IN

VACUUM AT 700° C FOR 5 MIN. (me volatile/gm in wt)

COMPOSITE NO.	(a) WT LOSS, %	(b) VOLATILE WT LOSS, %	VOLATILES me/g							(d) TOTAL me/g
			CO_2	CO	CH_4	HCN	C_6H_6	NH_3	H_2 (c)	
1	48.8 ± 2.1	0.8 ± 0.1	2.02 ± 0.53	0.36 ± 0.08	0.74 ± 0.04	0.24 ± 0.03	0.05 ± 0.01	-	1.63 ± 0.10	5.03 ± 0.18
2	48.8 ± 2.1	12.0 ± 0.8	1.54 ± 0.07	0.54 ± 0.09	1.26 ± 0.20	0.32 ± 0.03	0.06 ·	-	1.76 ± 0.32	5.64 ± 0.38
3	39.9 ± 2.2	12.9 ± 0.2	1.93 ± 0.14	0.50 ± 0.03	0.92 ± 0.24	0.28 ± 0.02	0.04 ± 0.01	-	2.16 ± 0.54	5.80 ± 0.40
4	41.9 ± 2.8	14.4 ± 0.6	2.22 ± 0.17	0.51	1.06 ± 0.02	0.26 ± 0.01	0.06 ± 0.01	-	1.60 ± 0.23	5.68 ± 0.05
5	42.0 ± 1.3	12.6 ± 0.1	1.89 ± 0.14	0.52 ± 0.05	0.30 ± 0.30	0.26 ± 0.01	0.042 ± 0.001		1.94 ± 0.40	5.61 ± 0.51
6	42.6 ± 1.1	12.4 ± 1.3	1.92 ± 0.20	0.42 ± 0.09	0.80 ± 0.03	0.22	0.062 · 0.005	-	1.72 ± 0.23	5.02 ± 0.52
7	35.8 ± 7.0	10.8 ± 1.8	1.81 ± 0.34	0.32 ± 0.09	0.26 ± 0.02	0.18 ± 0.03	0.04 ± 0.03	0.28 ± 0.28	0.16 ± 0.02	3.45 ± 0.31
8	43.6	11.6	1.95	0.22	0.59	0.12	0.053	-	0.62	3.56
9	20.4 ± 0.3	18.0 ± 0.1	3.53 ± 0.10	0.51 ± 0.06	0.08 ± 0.04	0.22 ± 0.02	-	0.21 · 0.12	-	4.22 ± 0.01

(a) WT LOSS, % = $\left[\dfrac{\text{(INITIAL WT SAMPLE)} - \text{(FINAL WT SAMPLE)}}{\text{(INITIAL WT SAMPLE)}} \right]$ (100)

(b) VOLATILE WT LOSS, % = $\left[\dfrac{\sum\limits_{i} \text{(MILLIEQUIVALENTS)}_i \text{(MOLECULAR WT)}_i}{\text{(INITIAL WT SAMPLE)} (100)} \right]$

(c) H_2 DETERMINED BY DIFFERENCE

(d) TOTAL me/g VOLATILES DETERMINED FROM: me/g = $\left[\dfrac{(P)(V)}{(R)(T)} \right] \left[\dfrac{1}{\text{INITIAL WT SAMPLE}} \right]$

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TABLE 5. – MAJOR VOLATILE PRODUCTS AT 23° C FROM THE PYROLYSIS OF COMPOSITES IN

VACUUM AT 700° C FOR 5 MIN. (mg volatile/gm in wt)

COMPOSITE NO.	QUANTITY[a]						
	CO_2	CO	CH_4	HCN	C_6H_6	NH_3	H_2
1	88.9	10.1	11.8	6.5	3.9		3.3
2	67.8	15.1	20.2	8.6	4.7		3.5
3	84.9	14.0	14.7	7.6	3.1		4.3
4	97.7	14.3	17.0	7.0	4.7		3.2
5	83.2	14.6	14.4	7.0	3.3		3.9
6	84.5	11.8	12.8	5.9	4.8		3.4
7	79.6	9.0	4.2	4.9	3.1	4.8	3
8	85.5	6.2	9.4	3.2	4.1		1.2
9	155.3	14.3	1.3	5.9		3.6	

[a] MILLIGRAMS OF VOLATILE COMPOUND AT 23 C PER GRAM OF
INITIAL SAMPLE

TABLE 6.- LIMITING OXYGEN INDEX FOR COMPOSITE COMPONENTS.

COMPOSITE NO.	COMPOSITE COMPONENT	LOI @ 23 C $O_2/(N_2 + O_2)$
1	PVF, PHENOLIC A/7581 GLASS, PHENOLIC B/120 GLASS	27
	AROMATIC POLYAMIDE-PAPER	32
	PHENOLIC A/7581 GLASS, PHENOLIC B/120 GLASS	26
	AVERAGE	28.3
2	PVF, PHENOLIC C/7581 GLASS, PHENOLIC D/120 GLASS	45
	AROMATIC POLYAMIDE-PAPER	32
	PHENOLIC C/7581 GLASS, PHENOLIC D/120 GLASS	32
	AVERAGE	36.3
3	PVF, PHENOLIC C/7581 GLASS, PHENOLIC C/120 GLASS	38
	AROMATIC POLYAMIDE-PAPER	32
	PHENOLIC E/120 GLASS (2 PLIES)	33
	AVERAGE	34.3
4	PVF, PHENOLIC E/7581 GLASS, PHENOLIC F/120 GLASS	47
	AROMATIC POLYAMIDE-PAPER	32
	PHENOLIC E/120 GLASS (2 PLIES)	30
	AVERAGE	35.3
5	PVF, PHENOLIC F/7581 GLASS, PHENOLIC F/120 GLASS	44
	AROMATIC POLYAMIDE-PAPER	32
	PHENOLIC F/120 GLASS (2 PLIES)	32
	AVERAGE	36
6	PVF, PHENOLIC G/7581 GLASS, PHENOLIC G/120 GLASS	74
	AROMATIC POLYAMIDE-PAPER	32
	PHENOLIC G/120 GLASS (2 PLIES)	36
	AVERAGE	47.3
7	BISMALEIMIDE/120 GLASS/POLYIMIDE	99
	AROMATIC POLYAMIDE-PAPER	32
	QUINONE DIOXIME FOAM	100
	BISMALEIMIDE/120 GLASS/POLYMIDE	99
	AVERAGE	82.5
8	PVF, EPOXY H/181E GLASS, EPOXY H/120 GLASS	29
	AROMATIC POLYAMIDE-PAPER	32
	EPOXY H/181 GLASS, EPOXY H/120 GLASS	28
	AVERAGE	29.6
9	BISMALEIMIDE/181E GLASS/POLYIMIDE	62
	BISMALEIMIDE/GLASS	58
	CARBON MICROBALLOONS/BISMALEIMIDE	85
	BISMALEIMIDE/181 GLASS/POLYIMIDE	62
	AVERAGE	66.7

TABLE 7.– SMOKE EVOLUTION FROM COMPOSITE PANELS (NBS SMOKE CHAMBER, 2.5 W/cm^2).

COMPOSITE NO.	SPECIFIC OPTICAL DENSITY (FLAMING CONDITION)			NO. OF TESTS
	D_s, 1.5 min	D_s, 4.0 min	D_m, maximum	
1	44.7 ± 13.3	51.7 ± 6.9	53.3 ± 8.9	3
2	3.9 ± 3.9	5.7 ± 4.1	8.6 ± 3.0	3
3	11.6 ± 0.9	13.6 ± 3.2	17.1 ± 6.4	3
4	7.1 ± 5.5	7.2 ± 5.3	8.4 ± 5.8	3
5	9.2 ± 8.2	12.2 ± 8.5	15.3 ± 8.5	3
6	8.8 ± 8.2	10.3 ± 7.3	16.0 ± 6.7	3
7	1.3 ± 1.0	4.5 ± 3.5	20.4 ± 4.7	2
8	48.3 ± 7.4	58.7 ± 6.2	59.1 ± 6.4	3
9	1.0 ± 0.7	4.9 ± 4.0	18.2 ± 7.9	4
(NONFLAMING CONDITION)				
1	11.7 ± 4.5	19.2 ± 3.1	20.2 ± 3.3	3
2	0.4 ± 0.2	0.7 ± 0.2	1.6 ± 0.2	3
3	1.0 ± 0.7	1.2 ± 0.7	2.0 ± 1.3	3
4	0.7 ± 0.1	0.7 ± 0.2	1.5 ± 0.2	3
5	1.2 ± 0.2	2.6 ± 0.3	5.2 ± 0.7	3
6	2.1 ± 0.8	2.2 ± 0.6	2.7 ± 0.7	3
8	2.6 ± 0.3	11.2 ± 1.1	19.0 ± 1.9	3

TABLE 8.- SMOKE EVOLUTION FROM COMPOSITE PANELS FROM TWO CHAMBERS (NBS SMOKE CHAMBER; 2.5 N/cm², FLAMING).

SPECIFIC OPTICAL DENSITY

COMPOSITE NO.	D_s, 1.5 min			D_s, 4.0 min			D_m, maximum		
	CHAMBER A	CHAMBER B	AVERAGE	CHAMBER A	CHAMBER B	AVERAGE	CHAMBER A	CHAMBER B	AVERAGE
1	44.7	—	44.7	51.7	—	51.7	53.3	—	53.3
2	3.9	10.5	7.2	5.7	15.5	10.6	8.6	19.4	14.0
3	11.6	12.7	12.2	13.6	15.9	14.8	17.9	17.4	17.6
4	7.1	11.7	9.4	7.2	13.9	10.6	8.4	16.1	12.2
5	9.2	16.7	13.0	12.2	20.7	16.4	15.3	22.8	19.0
6	8.8	13.3	11.0	10.3	16.6	13.4	16.0	17.3	16.6
7	1.3	—	1.3	4.5	—	4.5	20.4	—	20.4
8	48.3	57.6	53.0	58.7	57.5	58.1	59.1	58.3	58.7
9	1.0	—	1.0	4.9	—	4.9	18.2	—	18.2

TABLE 9.- RELATIVE TOXICITY OF PYROLYSIS PRODUCTS FROM COMPOSITE PANELS.

(1.0 g POWDERED SPECIMENS PYROLYZED AT 40 C/min TO 700 C;
4 SWISS ALBINO MICE IN 4.2 LITER EXPOSURE CHAMBER, 30 min EXPOSURE)

PANEL NO.	TEST NO.	TIME TO INCAPACITATION, min	TIME TO DEATH, min
1	1	18.1	28.31 · 1.67
	2	21.9	25.21 · 3.51
	3	16.3	25.83 · 1.02
	4	18.9	22.90 · 1.42
	MEAN	18.8	25.56 · 2.76
2	1	20.9	26.74 · 0.89
	2	21.0	24.90 · 0.11
	MEAN	21.0	25.82 · 1.13
3	1	19.0	24.52 · 0.69
	2	22.1	25.35 · 0.97
	MEAN	20.6	24.94 · 0.90
4	1	20.5	24.17 · 3.01
	2	19.3	23.48 · 0.31
	MEAN	19.9	23.82 · 2.01
5	1	20.3	26.18 · 1.83
	2	19.7	22.48 · 0.52
	MEAN	20.0	24.33 · 1.17
6	1	17.1	19.65 · 0.31
	2	20.9	22.90 · 0.96
	MEAN	19.0	21.28 · 0.63
7	1	22.8	27.40 · 1.46
	2	24.8	28.28 · 0.70
	MEAN	23.8	27.84 · 1.16
8	1	18.5	27.50 · 1.86
9	1	8.7	N.D.
	2	N.I.	N.D.

N.I. – NO INCAPACITATION OBSERVED
N.D. – NO DEATHS

TABLE 10.- SUMMARY OF COMPOSITE PROPERTIES (NO. 8, 9).

PROPERTY	COMPOSITE NO. 8	COMPOSITE NO. 9
BULK DENSITY RANGE kg/m^3 (ASTM D 1622)	90–96	109–113
FLATWISE TENSILE STRENGTH, AT 23°C KN/m^2 (ASTM C-307)	690	500–610
VERTICAL BURN TEST, FAA FAR 25.853	PASSES	PASSES
THERMAL CONDUCTIVITY AT 23°C $W \cdot cm/cm^2 \cdot °C$ (ASTM C-177-45)	$1.296 - 1.44 \times 10^{-3}$	4.932×10^{-4}
SMOKE DENSITY (NBS). (SPECIFIC OPTICAL DENSITY, FLAMING CONDITION, 2.5 W/cm^2	D_s 1.5 min.:53.0 D_s 4.0 min.:58.1 D_m :58.7	1.0 4.9 18.2
LIMITING OXYGEN INDEX, $O_2/(N_2+O_2)$. (ASTM D-2863)	EPOXY-FIBERGLASS:29 AROMATIC POLYAMIDE:32 COMPOSITE: 29.6	BISMALEIMDE-FIBERGLASS/POLYIMIDE:62 CARBON MICROBALLOONS, BISMALEIMIDE:85 BISMALEIMIDE-FIBERGLASS:58 COMPOSITE:66.7

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Table 10 – Concluded.

PROPERTY	COMPOSITE NO. 8	COMPOSITE NO. 9
RELATIVE TOXICITY OF PYROLYSIS PRODUCTS (1.0 g, 40°C/min to 700°C, 4 MICE IN 4.2 liter EXPOSURE CHAMBER. 30 min EXPOSURE) TIME TO INCAPACITATION; TIME TO DEATH (min)	18.5; 27.5 ± 1.86	>30; >30
FIRE ENDURANCE, NASA AMES T-3 TEST, TIME TO REACH BACK-FACE TEMP. OF 204°C AT FRONT FACE HEAT FLUX OF 10.4–11.9 W/cm^2	2 min. 20 sec	7 min. 45 sec
VOLATILES AT 23°C FROM PYROLYSIS IN VACUUM AT 700°C FOR 5 min Mg OF VOLATILE PER g OF INITIAL SAMPLE.	CO_2:85.5 CO : 6.2 CH_4: 9.4 HCN: 3.2 C_6H_6: 4.1 NH_3: – H_2 : 1.2	155.3 14.3 1.3 5.9 – 3.6 –
TGA, % WEIGHT REMAINING AT 700°C (N_2, 10°C/min).	51.0	73.5

DECORATIVE FILM

RESIN/GLASS
FACE SHEET

ADHESIVE FILM

HONEYCOMB
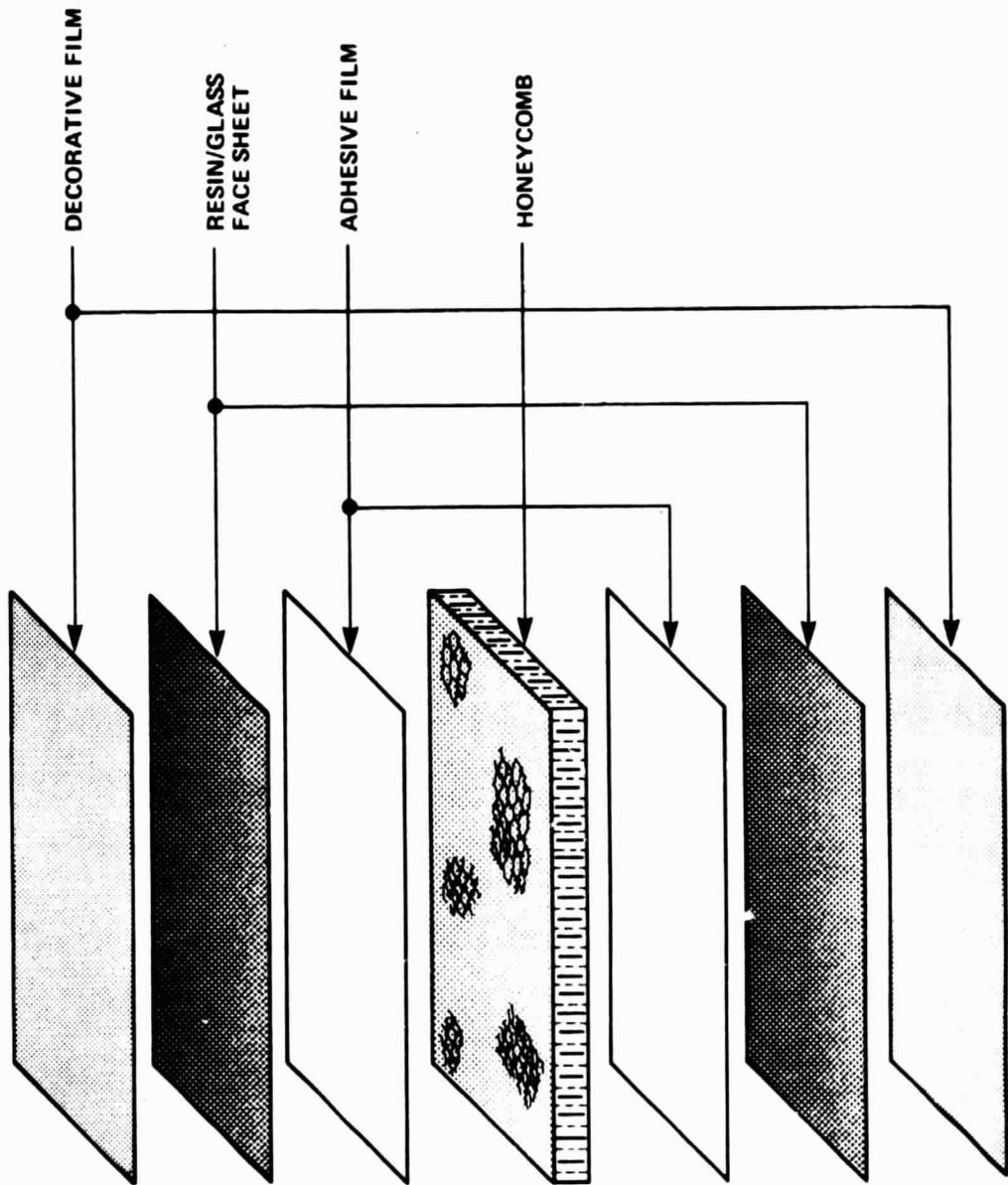
Figure 1.- Typical Composite Configuration of Aircraft Interior Panels.

COMPOSITES NO. 1–6

PHENOLIC

COMPOSITE NO. 8

DIGLYCIDYL ETHER
OF BISPHENOL A
CURED WITH
METHYLENE DIANILINE
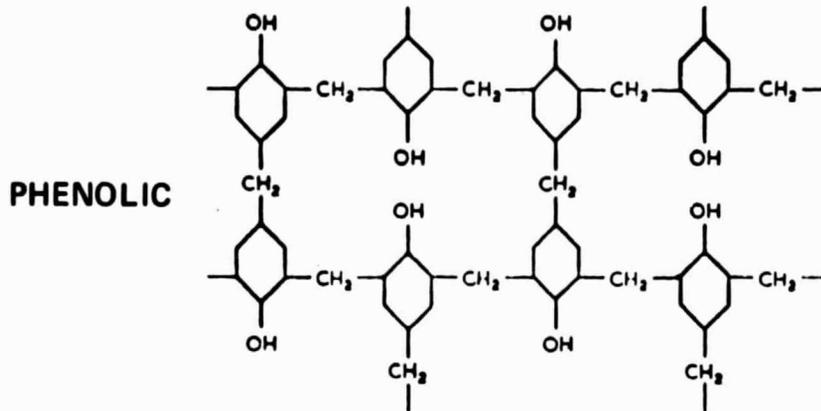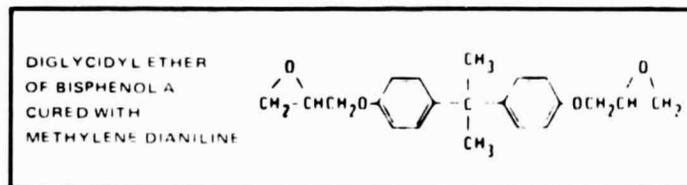
COMPOSITES NO. 7, 9

BISMALEIMIDE
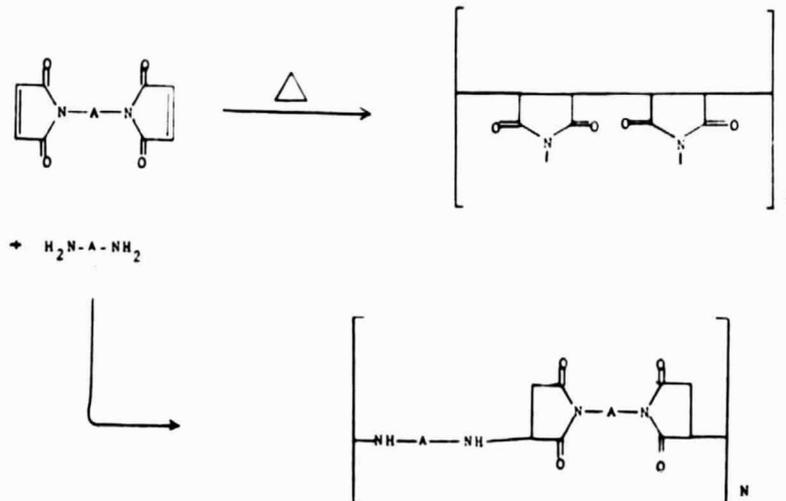
Figure 2.- Chemical Structure of Laminating Resins.

PREPARE CARBON MICROBALLOONS

- PHENOLIC MICROBALLOONS
- 4 hr @ 816 C
- COOL TO 23 C

DISPERSE IN SOLVENT

- BREAK UP CAKING
- DEAGGLOMERATE
- SCREEN

IMPREGNATE HONEYCOMB CORE

- VACUUM IMPREGNATE
- DRY 93° C
- WEIGH

APPLY POLYIMIDE BINDER

- DILUTE BISMALEIMIDE SOLUTION
- BRUSH
- CURE 2 hr @ 204° C

ASSEMBLE PANEL STRUCTURE

- POLYIMIDE ADHESIVE
- PRESS CURE
- 177° C
- POST CURE
- 2 hr @ 254° C

PREPARE BISMALEIMIDE SOLUTION

- 50% SOLUTION IN N-MP

PREPARE PREPREG

- B-STAGE
- RESIN CONTENT

CURE FACESHEETS

- VACUUM BAG
- BLEEDER CLOTH
- 1 hr @ 177° C

Figure 3.- Fabrication Process for Composite No. 9.
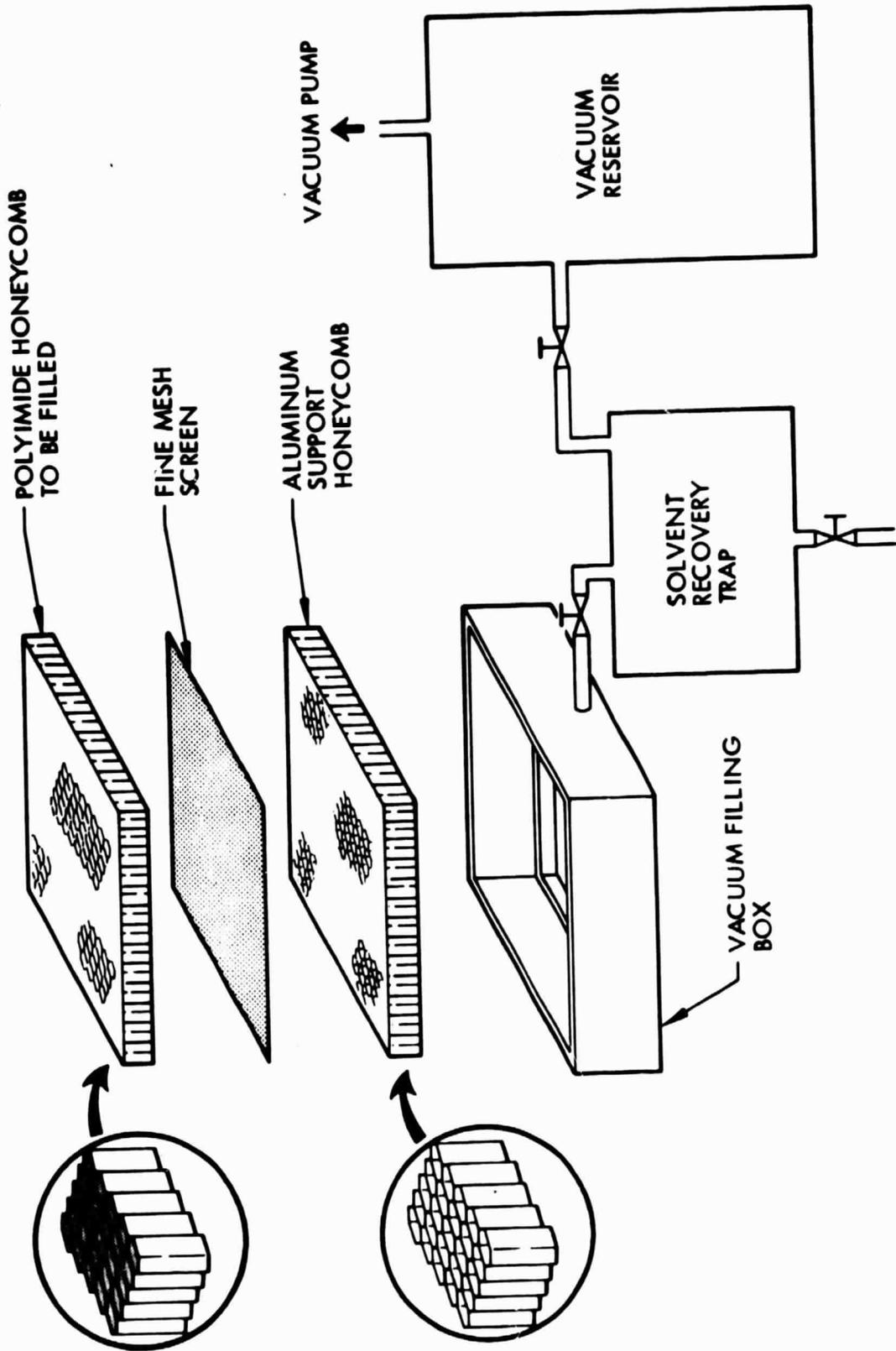
Figure 4.- Honeycomb Core Impregnation Equipment.
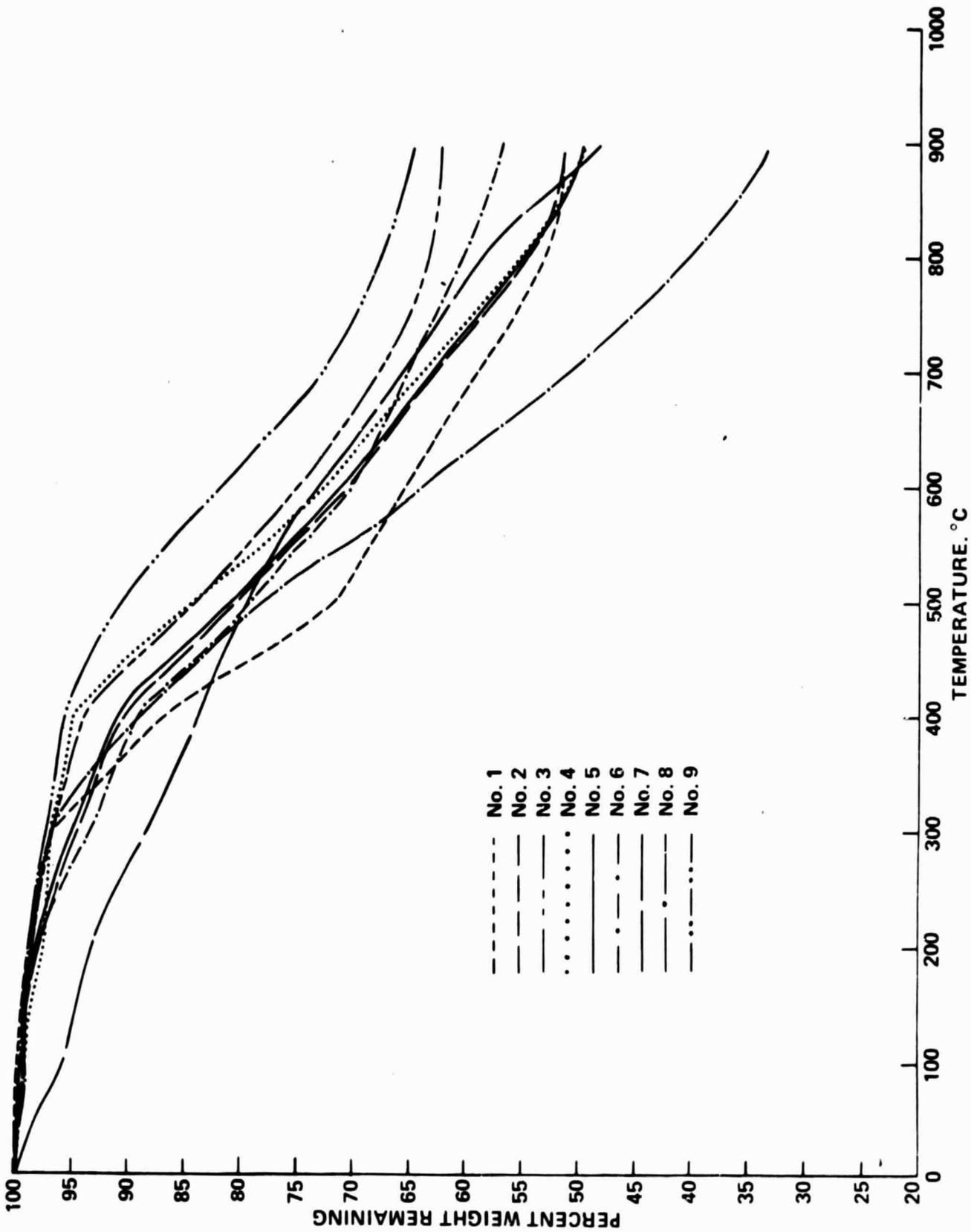
Figure 5.- Dynamic Thermograph of Composites 1-9 (heating rate 10° C/min, N_2).
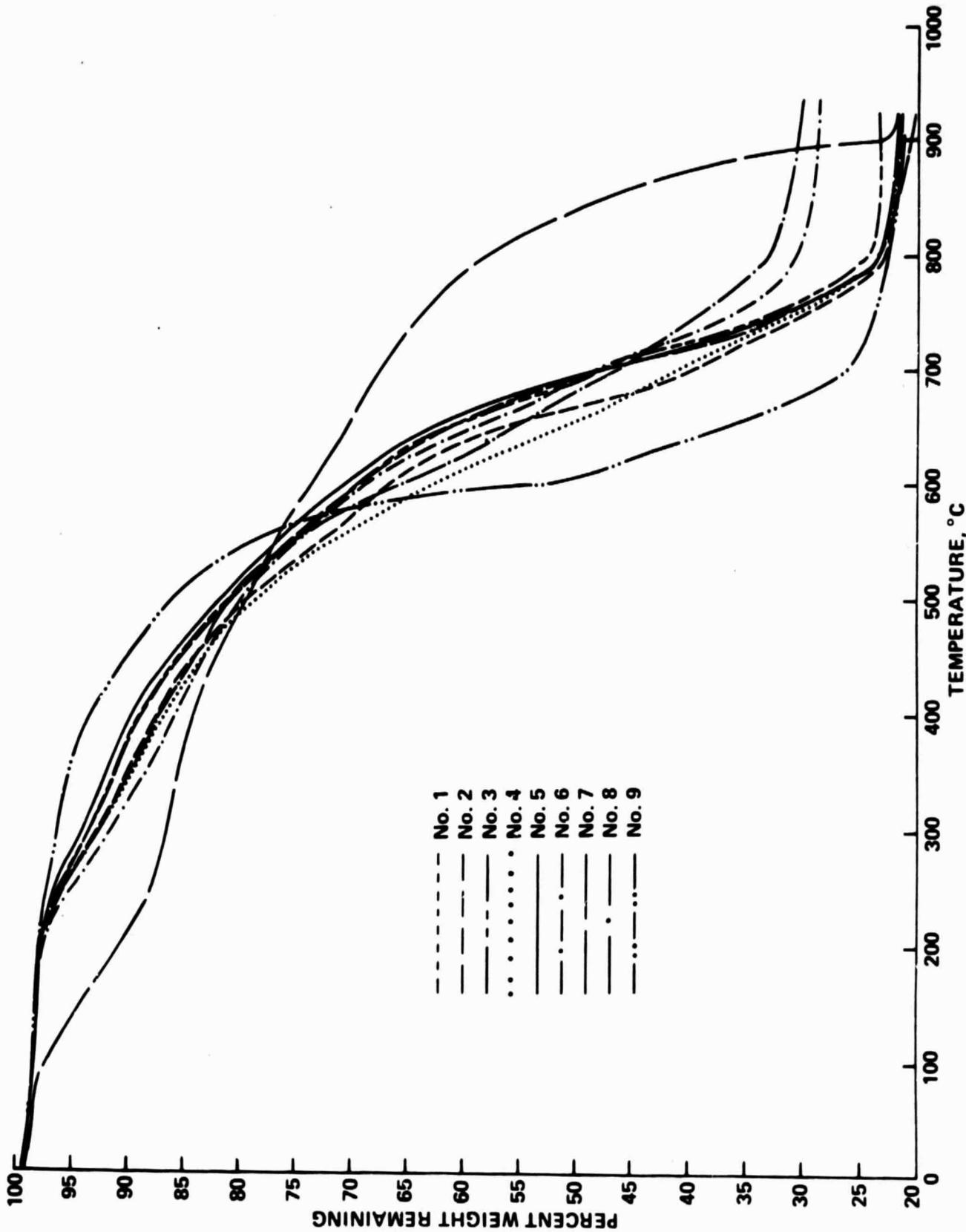
Figure 6.- Dynamic Thermograph of Composites 1-9 (heating rate 10° C/min, air).
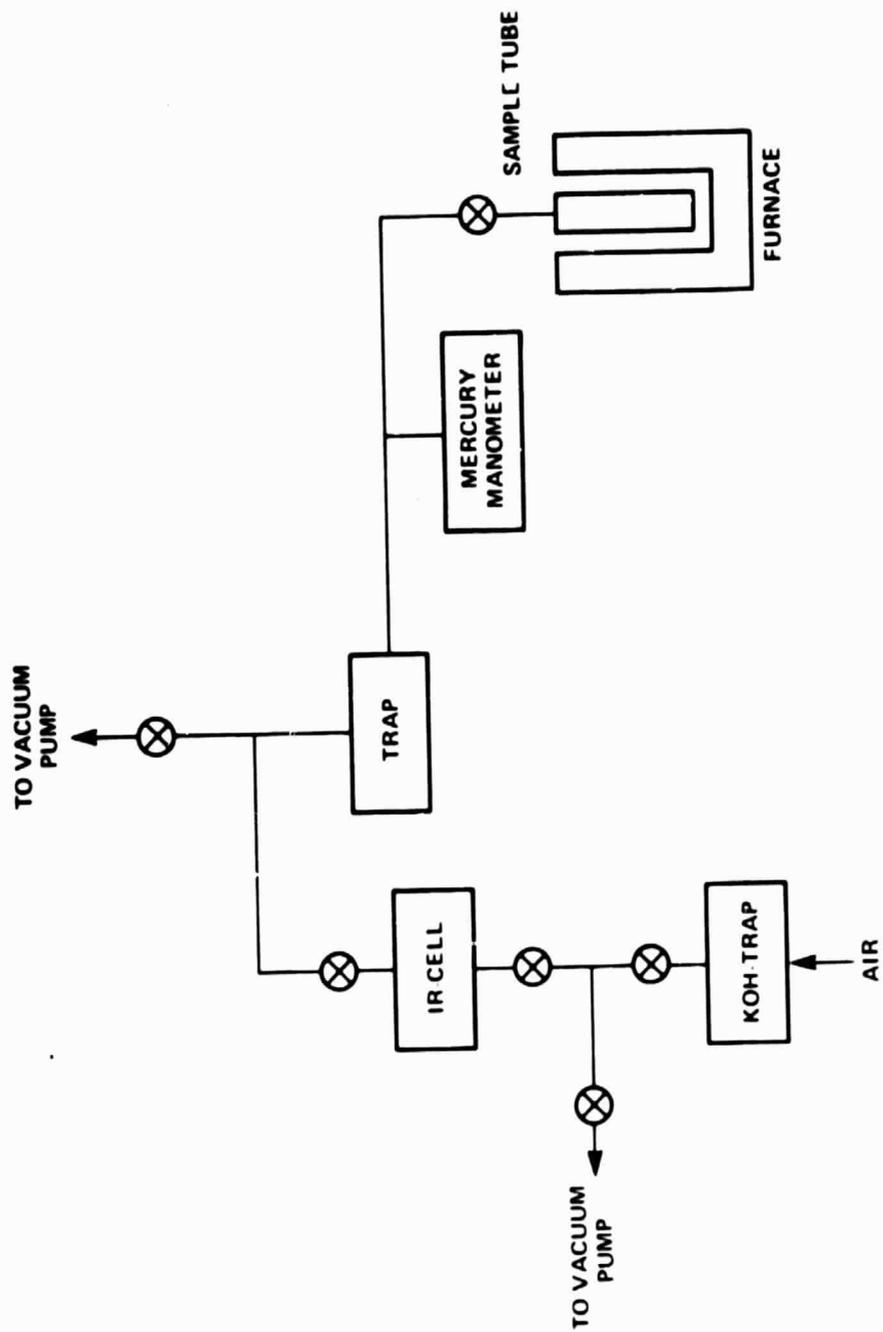
Figure 7.- Apparatus for pyrolysis of materials.

COMPOSITE SPECIMEN

THERMOCOUPLES

HEAT RESISTANT GLASS

MIRROR

JP-4 FUEL

OIL BURNER

FIRE BRICK LINED COMBUSTION CHAMBER

TYPICAL HEAT FLUX AT Ⓐ 10.4–11.9 W/cm^2

TYPICAL TEMPERATURE AT Ⓐ 700°–900°C
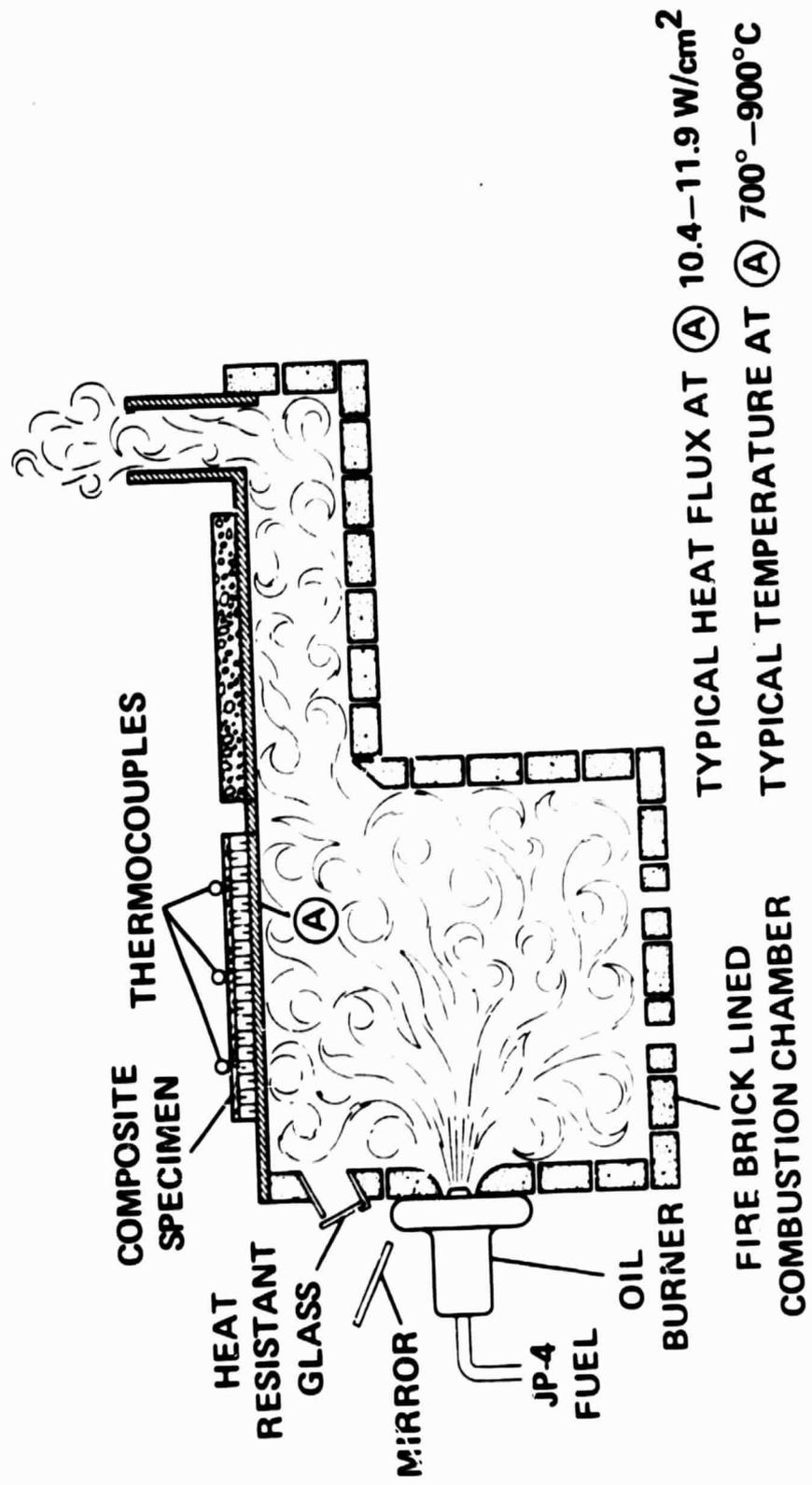
Figure 8. - NASA–Ames T-3 Thermal Efficiency Apparatus.
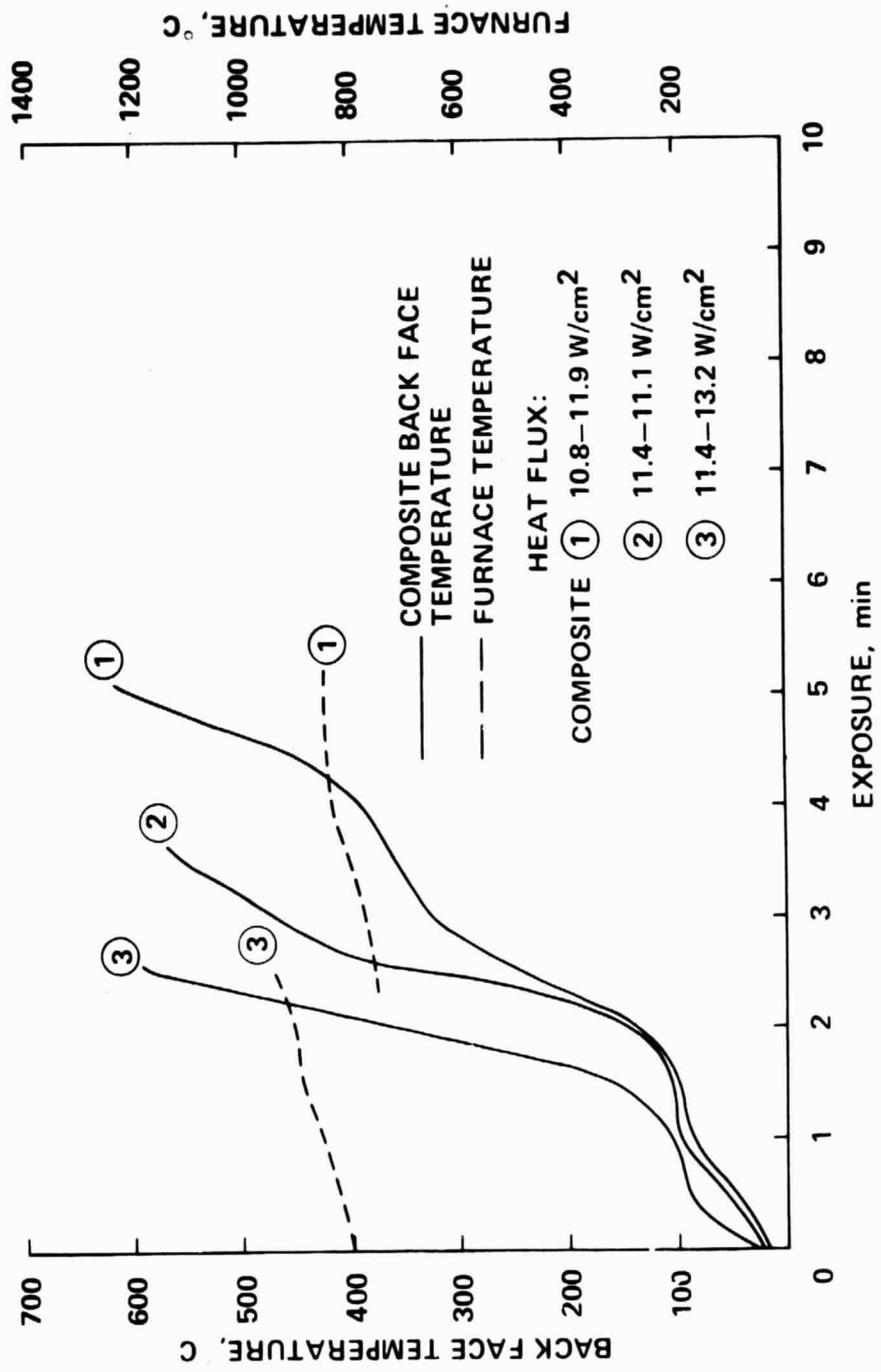
Figure 9.- Thermal Efficiency of Composites: No. 1-3.
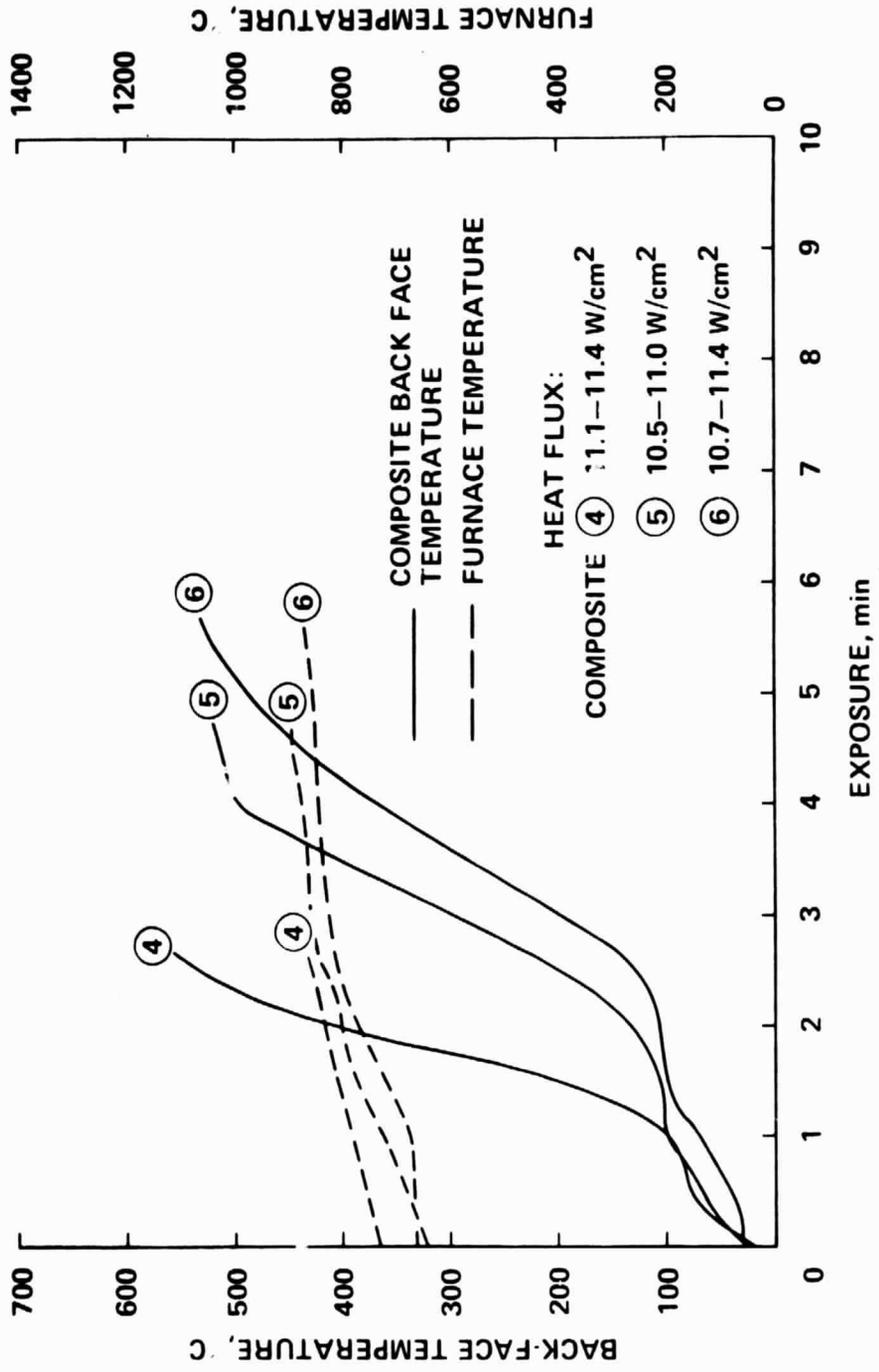
Figure 10. Thermal Efficiency of Composites: No. 4-6.
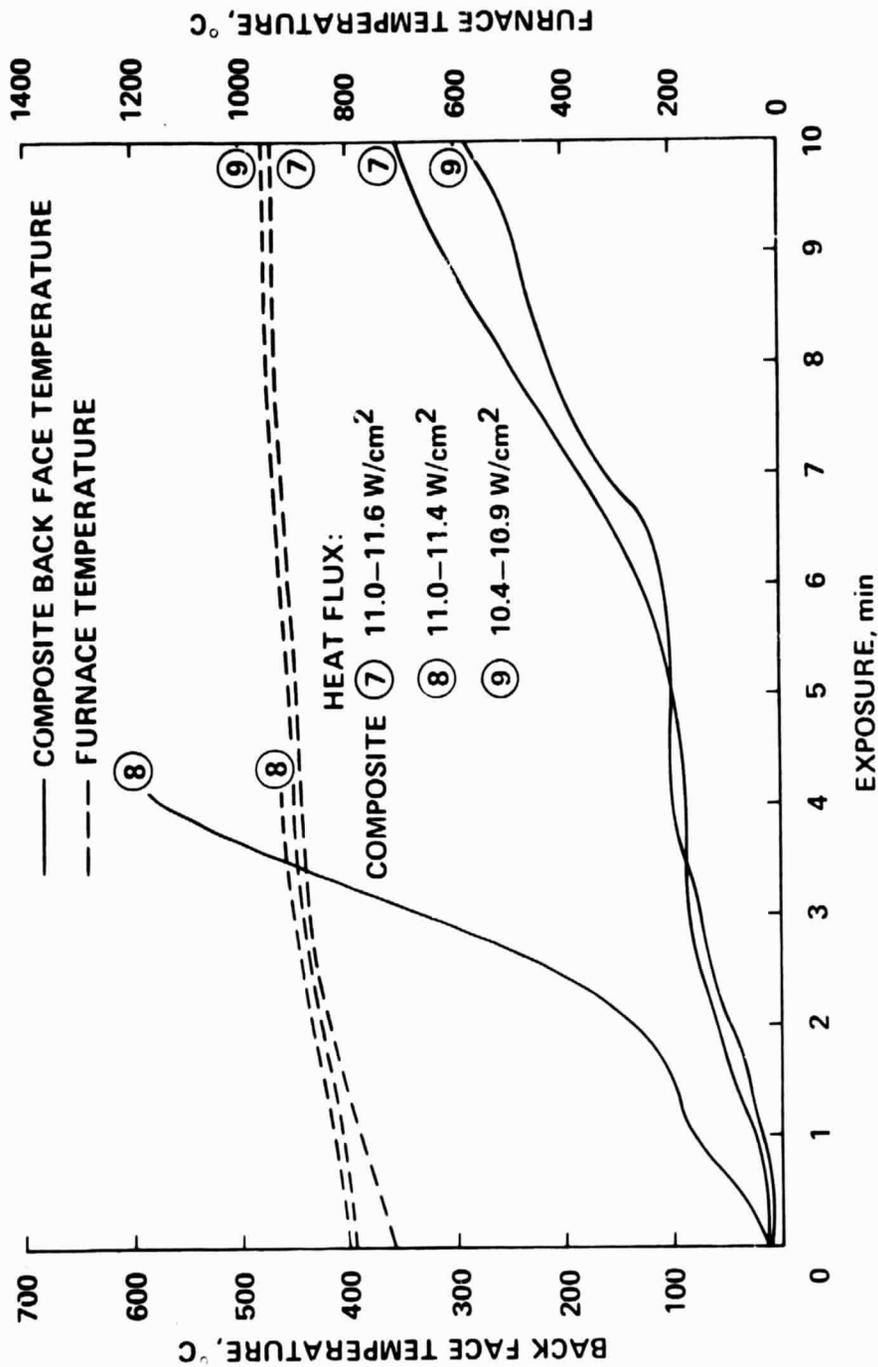
Figure 11. Thermal Efficiency of Composites: No. 7-9.
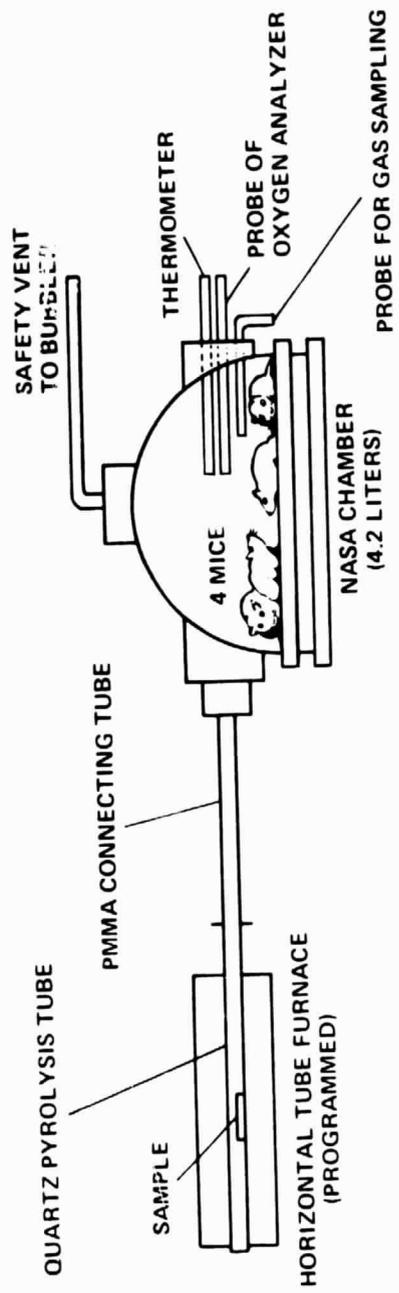
Figure 12. Pyrolysis Toxicity Apparatus.
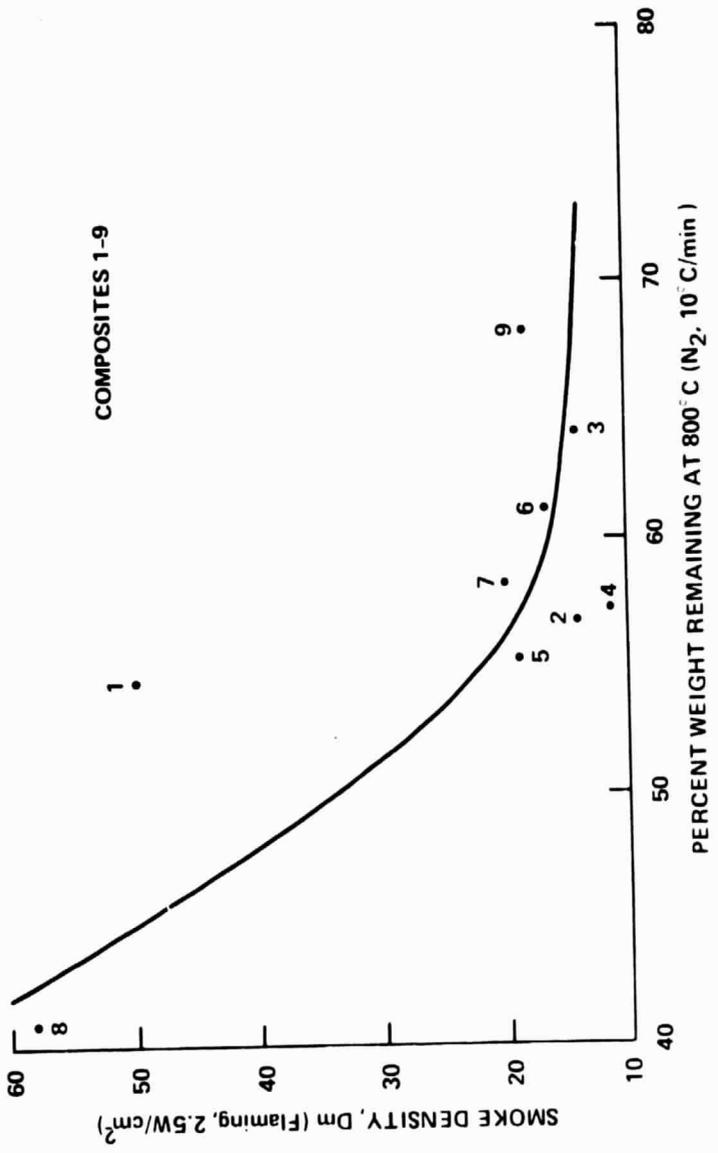
Figure 13. Effect of Char Yield of Composites on Smoke Evolution.
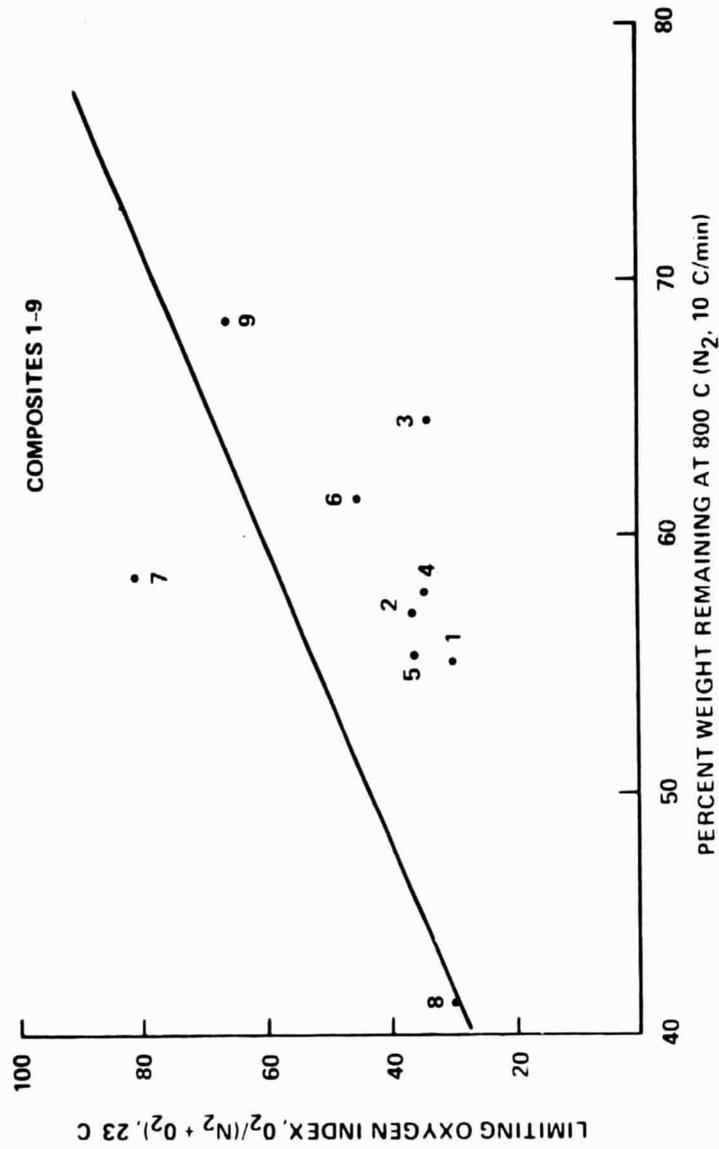
Figure 14. Effect of Char Yield of Composites on Limiting Oxygen Index.